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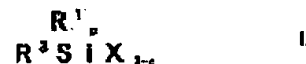
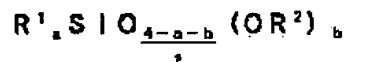
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(54) PRIMER COMPOSITION AND BONDING

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain the subject composition capable of firmly bonding and integrating room temperature curing polyisobutylene-based polymer composition with various kinds of substrates such as glass by including a specific organopolysiloxane resin, etc.

SOLUTION: This composition comprises (A) 100 pts.wt. of an organopolysiloxane resin containing silicon atom-binding hydroxyl groups or silicon atom-binding alkoxy groups represented by average unit formula I [R1 is a monovalent hydrocarbon; R2 is H or an alkyl; (a) is 0.8-1.8; and (b) is a value so as to provide the number of the silicon atom-binding hydroxyl groups or the silicon atom-binding alkoxy groups in one molecule with ≥1], (B) 20-400 pts.wt. of an amino group-containing organosilane represented by formula II [R3 is a monovalent hydrocarbon containing at least one amino group; X is a hydrolyzable group; and (c) is 0 or 1] or its partial hydrolyzate, (C) 20-400 pts.wt. of organotitanic esters (e.g. tetraisopropyl titanate) and (D) an optional amount of an organic solvent (e.g. benzene).



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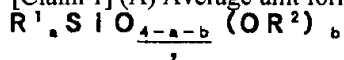
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 CLAIMS

[Claim(s)]

[Claim 1] (A) Average unit formula. [Formula 1]



(-- R1 is a univalent hydrocarbon group (however, an ARUKENIRU machine is removed) among a formula, R2 is a hydrogen atom or an alkyl group, a is 0.8-1.8, and b is a value from which the number of the silicon atomic-union hydroxyl group in 1 molecule or silicon atomic-union alkoxy groups becomes one or more pieces Organopolysiloxane resin containing the silicon atomic-union hydroxyl group or silicon atomic-union alkoxy group expressed with) The 100 weight sections and (B) general formula. [Formula 2]



(R1 is the same as the above among a formula, R3 is a monovalent hydrocarbon radical containing at least one amino group, X expresses the basis which can be hydrolyzed and c is the integer of 0 or 1.) The 20 - 400 weight section and (C) organic titanate ester The 20 - 400 weight section and (D) organic solvent Primer constituent for pasting up a room-temperature-curing nature polyisobutylene system polymer constituent on various base materials characterized by the bird clapper from an arbitrary dose.

[Claim 2] How to paste up the room-temperature-curing nature polyisobutylene system polymer constituent characterized by contacting a room-temperature-curing nature polyisobutylene system polymer constituent, and stiffening it after making a base-material front face apply and dry the primer constituent of a claim 1 on various base materials.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the adhesion method of the primer constituent with which a room-temperature-curing nature polyisobutylene system polymer constituent is pasted [want / to make it] up on various base materials, such as glass and a metal, and a room-temperature-curing nature polyisobutylene system polymer constituent in more detail about a primer constituent and the adhesion method.

[0002]

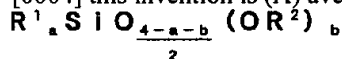
[Description of the Prior Art] Conventionally, many things are proposed as a room-temperature-curing nature polymer constituent. Also in these, since a room-temperature-curing nature polyisobutylene system polymer constituent has a mechanical property, weatherability, thermal resistance, and resistance to contamination, it attracts attention (refer to JP,4-69659,B, JP,6-93148,A, and JP,1-170658,A). However, this kind of room-temperature-curing nature polyisobutylene system polymer constituent was inferior to the adhesive property over various base materials, such as glass and a metal, and had the trouble that it could not be used, depending on the use.

[0003]

[Problem(s) to be Solved by the Invention] In order to cancel the above-mentioned trouble, as a result of inquiring wholeheartedly, when using the specific primer constituent, this invention persons found out carrying out the adhesion unification of a room-temperature-curing nature polyisobutylene system polymer constituent and the various base materials firmly, and reached this invention. That is, the purpose of this invention is to offer the method of being in offering the primer constituent for pasting up a room-temperature-curing nature polyisobutylene system polymer constituent to an extensive base material, and pasting up a room-temperature-curing nature polyisobutylene system polymer constituent on various base materials.

[Means for Solving the Problem]

[0004] this invention is (A) average unit formula. [Formula 3]



(-- R1 is a monovalent hydrocarbon radical (however, an alkenyl machine is removed) among a formula, R2 is a hydrogen atom or an alkyl group, a is 0.8-1.8 and b shows the value from which the number of the silicon atomic-union hydroxyl group in 1 molecule or silicon atomic-union alkoxy groups becomes one or more pieces Organopolysiloxane resin containing the silicon atomic-union hydroxyl group or silicon atomic-union alkoxy group expressed with) The 100 weight sections and (B) general formula. [Formula 4]



(R1 is the same as the above among a formula, R3 is a monovalent hydrocarbon radical containing at least one amino group, X expresses the basis which can be hydrolyzed and c is the integer of 0 or 1.) The 20 - 400 weight section and (C) organic titanate acid ester The 20 - 400 weight section and (D) organic solvent It is related with the primer constituent for pasting up a room-temperature-curing nature polyisobutylene constituent on various base materials characterized by the bird clapper from an arbitrary dose.

[0005] The organopolysiloxane resin of the (A) component used for this invention is a component used as the subject of the primer constituent of this invention, and in the primer constituent of this invention, an adhesive property and in order to give especially a waterproof adhesive property, it needs to have one or more hydroxyl groups or alkoxy groups in 1 molecule. For the inside of an upper formula, and R1, such a (A) component is the monovalent hydrocarbon radical (however) of the substitution which is what replaced aryl groups and these hydrogen atoms, such as alkyl group; phenyl groups, such as a methyl group, an ethyl group, and a propyl group, and a tolyl group, by the halogen atom, the cyano atom, etc., and is illustrated, or not replacing. Express and R2 expresses the alkyl group illustrated by the hydrogen atom or the methyl group, the ethyl group, the propyl group, etc. an alkenyl machine -- not containing -- a is an average of 0.8-1.8, and b shows the value from which the one or more number of the hydroxyl group combined with the silicon atom in 1 molecule or alkoxy groups

becomes three or more pieces preferably. Usually, one sort or two sorts or more of mixture is understood an added water part under mixture of water or water, and an organic solvent, and the organopolysiloxane containing this hydroxyl group or alkoxy group is obtained by removing the hydrochloric acid of the crawl silane or alkoxy silane which has 0.8-1.8 monovalent hydrocarbon radicals per silicon atom which carries out a byproduction, alcohol, etc., respectively.

[0006] The amino-group content silane of the (B) component used for this invention or its partial hydrolysate gives the adhesive property over an extensive base material to the primer constituent of this invention. R1 of this (B) component is the same as that of what was mentioned above among an upper formula, R3 is a monovalent hydrocarbon radical containing at least one amino group, and an aminomethyl machine, beta-aminoethyl machine, gamma-aminopropyl machine, gamma-(2-aminoethyl) aminopropyl machine, a gamma-[2-(2-aminoethyl) aminoethyl]-aminopropyl machine, etc. are specifically illustrated. X is the basis which can be hydrolyzed and alkoxy-group; dialkylamino machine [, such as a methoxy machine and an ethoxy basis,], ketoxime machine, N, and N-dialkyl aminoxy etc. is specifically illustrated. As an example of this component, aminomethyl trimethoxysilane, aminomethyl triethoxysilane, gamma-aminopropyl trimethoxysilane, gamma-aminopropyl triethoxysilane, a gamma-amino pro PIRUTORI (methylethyl ketoxime) silane, a gamma-amino pro PIRUTORI (N and N-diethyl friend NOKISHI) silane, gamma-(2-aminoethyl) aminopropyl trimethoxysilane, etc. are mentioned. Moreover, since an effect with the same said of the partial hydrolysate of these silanes is shown, it is usable. The range of the 20 - 400 weight section is suitable for the blending ratio of coal of this (B) component to the (A) component 100 weight section.

[0007] It carries out the work which gives an adhesive property to this invention constituent while the organic titanate-acid ester of the (C) component used for this invention acts as a catalyst for stiffening the primer constituent of this invention. As an example of this component (C), titanium chelate compounds, such as tetrapod isopropyl titanate, a tetrapod normal butyl titanate, tetrapod (2-ethylhexyl) titanate and these partial hydrolysis condensates, diisopropyl CHITAMBISU acetyl acetate, and diisopropyl titanium bis-ethylacetoacetate, are mentioned. The range of the 20 - 400 weight section is suitable for the blending ratio of coal of this component to the (A) component 100 weight section.

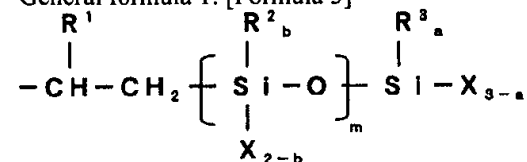
[0008] The organic solvent of the (D) component used for this invention dissolves the primer constituent of this invention, and is chosen in consideration of the wettability which is in charge of the application work as the solubility [as opposed to / are and / (A) - (C) component] and primer for raising the solvent nature to the various base materials of this invention constituent, and evaporativity. As an example of such an organic solvent, benzene, toluene, a xylene, a hexane, a heptane, a trichloroethylene, a par chloroethylene, a methanol, ethanol, an isopropanol, a butanol, etc. are mentioned. These organic solvents are used as independent or two or more sorts of partially aromatic solvents. Although such an organic solvent is not necessarily needed when the viscosity of the mixture of (A) - (C) component is the viscosity range which can be enough used as a primer constituent. In many cases, the constituent of this invention is dissolved, and it is advantageous to be prepared and used for the viscosity and concentration suitable for application work actual as a primer, and it is usually used within the limits of the 1 - 10,000 weight section to the (A) component 100 weight section.

[0009] Although the primer constituent of this invention is easily obtained only by only mixing said (A) component - (D) component uniformly, further, in addition to these, it is the range which does not bar the purpose of this invention, and can blend suitably well-known coloring agents, such as inorganic bulking agent; red ochre, such as other alkoxy-group content silane; fumed silicas and colloidal silica, titanium oxide, and carbon black, etc. conventionally.

[0010] Especially the kind etc. is not limited that the room-temperature-curing nature polyisobutylene system polymer constituent pasted up with the primer constituent of this invention should just be a constituent which makes a principal component the isobutylene system polymer obtained by carrying out the polymerization of the isobutylene system monomer, and is hardened at a room temperature. As this polyisobutylene system polymer constituent, there is a constituent as shown below, for example.

** The room-temperature-curing nature constituent which makes a principal component the isobutylene system polymer which has the reactant machine shown in a molecule end by at least one following general formula 1 (refer to JP,4-69659,B).

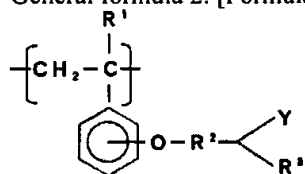
General formula 1. [Formula 5]



the inside of a formula, and R1 -- a hydrogen atom, the alkyl group of carbon numbers 1-8, the aryl group of carbon numbers 6-20, or the aralkyl machine of carbon numbers 7-20 -- Each of R2 and R3 The alkyl group of carbon numbers 1-20, the aryl group of carbon numbers 6-20, The aralkyl machine or (R)3SiO of carbon numbers 7-20 - (R is the univalent hydrocarbon group of carbon numbers 1-20) It is the trio luganot siloxy machine shown. being the same -- differing -- **** -- X which may differ may be a hydroxyl group or a adding-water resolvability machine, it may be the same, when two or more pieces join together, it may be the same, and a which may differ is [0, 1, or 2m of 0, 1, 2, or 3b] the integer of 0, or 1-18

** The room-temperature-curing nature constituent which consists of an isobutylene system polymer which has the silicon atomic-union hydroxyl group or adding-water resolvability machine shown by the following general formula 2, and a silanol condensation catalyst (refer to JP,6-93148,A).

General formula 2. [Formula 6]



(R1 shows a hydrogen atom, a methyl group, or an ethyl group among a formula.) R2 shows the divalent organic machine of carbon numbers 1-20, and R3 shows a hydrogen atom or the univalent organic machine of carbon numbers 1-10. Y shows a reactant silicon machine.

** The polyisobutylene polymer constituent which becomes an end or a side chain from the polyisobutylene system polymer and silicon atomic-union hydrogen atom content ORGANO hydrogen polysiloxane which have an allyl group, and the catalyst for a hydrosilylation reaction [refer to Polymer Bulletin, 18, and 463 (1987)].

[0011] What is necessary is to contact a room-temperature-curing nature polyisobutylene system polymer constituent to this primer application side, and just to make it harden first, in order to paste up the above room-temperature-curing nature isobutylene system polymer constituents on various base materials using the primer constituent of this invention, after making various base-material front faces apply and dry the primer constituent of this invention.

[0012]

[Example] Hereafter, this invention is explained based on an example. In addition, in the following examples, the section expresses the weight section, and Me expresses a methyl group.

[0013]

[Example 1] (CH3) It consisted of 40 mol % of 2SiO₂/2 units, and 60 mol % of CH₃SiO₃/2 units, and the primer constituent 1, the primer constituent 2, and the primer constituent 3 of the composition which mixes the 50-% of the weight xylene solution of the methyopolysiloxane resin whose hydroxyl-group content is 1 % of the weight, gamma-(2-aminoethyl) aminopropyl trimethoxysilane, methyl trimethoxysilane, tetrapod butoxysilane, and a normal hexane, and is shown in Table 1 were prepared. These primer constituents were applied to float sheet glass (product made from Japanese Test-panel Industry), and the aluminum plate (JIS product made from H4000 A6063 S Japan Test-panel Industry), respectively, and at the room temperature, it was left for 30 minutes and was air-dry. Next, after applying a room-temperature-curing nature polyisobutylene system polymer constituent on this primer application side and leaving it for seven days under conditions of the temperature of 20 degrees C, and 50% of humidity, a himself was recuperated for seven days and it was made to harden under 50 degrees C and conditions of 50% of humidity. Here, the room-temperature-curing nature polyisobutylene polymer constituent was a constituent which mixed uniformly the isobutylene polymer 100 section which has two -Si (CH₃) (OCH₃) in chain both ends, the water 0.5 section, and the toluene 50 section, and was obtained. It is JIS about the **** adhesive property of the obtained specimen. It measured according to the A5758 structural sealing material. Moreover, after carrying out the dipping of this specimen to 50-degree-C warm water for seven days, the **** adhesive property was measured like the above. These results were shown in the 2nd table as an initial adhesive property and a waterproof adhesive property, respectively. In addition, such adhesive measured value showed the cohesive failure (the polyisobutylene polymer hardened material fractured.) by O, and showed interfacial peeling (a polyisobutylene polymer hardened material did not fracture but it exfoliated from the base material.) by X. In the above, the primer constituent 4 shown in Table 1 like the above was prepared except having not carried out addition combination of the methyopolysiloxane resin for comparison. Moreover, the primer constituent 5 shown in Table 1 like the above was prepared except having not carried out addition combination of the N-(2-aminoethyl) aminopropyl trimethoxysilane. The property of these primer constituents was measured like the above, and those measurement results were written together to Table 2.

[0014]

[Table 1]

組成物 NO	本発明			比較例	
	1	2	3	4	5
メチルポリシロキサン樹脂溶液 (部)	2.5	5.0	2.5	0.0	2.5
γ-(2-アミノエチル)-アミノプロピルトリメトキシシラン (部)	2.5	2.5	1.25	2.5	0.0
メチルトリメトキシシラン (部)	0.0	0.0	1.25	0.0	2.5
テトラブトキシシラン (部)	2.5	2.5	2.5	2.5	2.5
ノルマルヘキサン (部)	92.5	90.0	92.5	95.0	92.5

[0015]

[Table 2]

組成物 NO		本発明			比較例		
		1	2	3	4	5	プライマ -無し
ポ-トガラス	初期接着性	○	○	○	×	○	×
	耐水接着性	○	○	○	×	×	×
アルミニウム板	初期接着性	○	○	○	×	×	×
	耐水接着性	○	○	○	×	×	×

[0016]

[Effect of the Invention] Since the primer constituent of this invention consists of a (A) component - (D) component and the organic titanate ester of the hydroxyl group of the (A) component or an alkoxy-group content organopolysiloxane resin, the amino-group content organosilane of the (B) component or its partial hydrolysate, and the (C) component is contained especially, it has the feature that it is possible to paste up firmly a room-temperature-curing nature polyisobutylene system polymer constituent on various base materials.

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CLAIMS

[Claim(s)]

[Claim 1] (A) The primer constituent characterized by consisting of a tin (IV) compound and (E) organic solvent as the hydroxyl group combined with the silicon atom or an alkoxy-group content organopolysiloxane resin, (B) amino-group content silane compound, (C) alkoxy silyl machine content isobutylene system polymer, and a (D) catalyst.

[Claim 2] The inside of the primer constituent whole quantity, the primer constituent according to claim 1 which are (Component A) 0.5-20 % of the weight, (Component B) 0.1-10 % of the weight, and (Component C) 0.5-30 % of the weight.

[Claim 3] The primer constituent according to claim 1 or 2 whose amount of a catalyst (D) is 0.1 - 10 weight section to the total quantity 100 weight section of component (A) - (C).

[Claim 4] The primer constituent according to claim 1, 2, or 3 whose weight ratios of a tin (IV) compound and organic titanate ester organic titanate ester is used together to a tin (IV) compound, and are 9:1-3:7.

[Claim 5] The claim 1 for polyisobutylene system sealing materials, or the primer constituent of any one publication of four.

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